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Compression and Instrumented Indentation Measurements on Biomimetic Polymers

by Thomas F. Juliano, Paul Moy, Tusit Weerasooriya,
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ARL-RP-135 September 2006

A reprint from the *Proceedings of the Society for Experimental Mechanics Conference*,
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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED (From - To)
September 2006	Reprint	August 2005–February 2006

4. TITLE AND SUBTITLE
Compression and Instrumented Indentation Measurements on Biomimetic Polymers

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
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5d. PROJECT NUMBER
622105.AH7G

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
U.S. Army Research Laboratory
ATTN: AMSRD-ARL-WM-MA
Aberdeen Proving Ground, MD 21005-5069

8. PERFORMING ORGANIZATION REPORT NUMBER
ARL-RP-135

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES
A reprint from the *Proceedings of the Society for Experimental Mechanics Conference*, St. Louis, MO, 6 June 2006.
*National Institute of Standards and Technology, Building and Fire Research Division, 100 Bureau Dr., Gaithersburg, MD 20899

14. ABSTRACT
The mechanical response of living tissue is important to understanding the injury-risk associated with impact events. Unfortunately, currently used materials are not optimal surrogates and present several experimental challenges. Bulk measurement techniques, such as compression and shear tests, do not necessarily represent the type and rate of loading experienced in an actual impact event. Indentation testing may induce surface loading at stress states and strain rates not available to bulk measurement equipment. In this work, a ballistic gelatin and two styrene-isoprene triblock copolymer gels are tested and compared using both macro-scale and micro-scale measurements. A methodology is presented to conduct instrumented indentation experiments with a flat punch on materials with a modulus far below 1 MPa. The synthetic triblock copolymer gels are shown to be much easier to test than the ballistic gelatin. Compared to ballistic gelatin, both copolymer gels were found to have a greater degree of thermal stability. All of the materials exhibit strain-rate dependence, although the magnitude of dependence is a function of the loading rate and testing method.

15. SUBJECT TERMS
indentation, polymers, gels

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Thomas Juliano
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL	14	19b. TELEPHONE NUMBER (*Include area code*) 410-306-1906

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ABSTRACT
The mechanical response of living tissue is important to understanding the injury-risk associated with impact events. Unfortunately, currently used materials are not optimal surrogates and present several experimental challenges. Bulk measurement techniques, such as compression and shear tests, do not necessarily represent the type and rate of loading experienced in an actual impact event. Indentation testing may induce surface loading at stress states and strain rates not available to bulk measurement equipment. In this work, a ballistic gelatin and two styrene-isoprene triblock copolymer gels are tested and compared using both macro-scale and micro-scale measurements. A methodology is presented to conduct instrumented indentation experiments with a flat punch on materials with a modulus far below 1 MPa. The synthetic triblock copolymer gels are shown to be much easier to test than the ballistic gelatin. Compared to ballistic gelatin, both copolymer gels were found to have a greater degree of thermal stability. All of the materials exhibit strain-rate dependence, although the magnitude of dependence is a function of the loading rate and testing method.

INTRODUCTION
Blunt trauma may have significant detrimental effects on thoracic organs, and understanding these effects is critical for the design of protective equipment used in the automotive, body armor, and sports industries, among others. Experiments using cadavers, instrumented dummies, and synthetic biomimetic materials assess the effects of extreme pressure and velocity on soft tissue. Standard tests and surrogate materials have been developed to evaluate and rank protective equipment [1]. Recent work to better understand how impact forces are transmitted through soft tissue have led to the fabrication of instrumented surrogate torsos composed of modified ballistic gelatin (BG) [2] or modified silicones [3]. Often lacking is the proper match of the surrogate mechanical properties to soft tissue properties. A closer pairing of the acoustic and mechanical properties of a well-defined synthetic material to natural tissue can improve the data collected from the surrogates, ultimately improving the ability to model impact damage.

Naturally derived BG has been most widely used as a tissue surrogate to develop a wound profile that provides limited injury analysis [4, 5]. For over 40 years, BG has been a standard test medium for evaluating the effects of ballistics and firearms on soft tissue [6, 7]. Previous research has shown that the elastic modulus of soft tissue varies from (25 to 300) kPa [8], with the modulus of BG being approximately (100 to 150) kPa [9, 10]. However, challenges in using BG as a standard test medium are significant. The mechanical properties, for example, are related to the concentration of water in the gel, therefore water evaporation dramatically alters the mechanical properties. Naturally derived gelatin is polydispersed and can have a variable molecular weight distribution that can lead to inconsistent gelatin properties from batch to batch. Finally, while a properly formed natural gelatin is a homogenous material, procedural differences exist with regard to gelatin processing and testing [2, 7-10]. These differences lead to gels of differing mechanical properties and make comparisons between researchers difficult.

Triblock copolymers possessing an A-B-A polymer structure and dissolved in a solvent that is selective for block B are potential candidates as surrogate materials. The elastic modulus of these gels is primarily determined by the

molecular weight of the B block (or molecular weight between entanglements) and the polymer concentration. By adjusting the concentration of polymer in the gel or the molecular weight of the B blocks, the elastic modulus of the gels can be tailored to mimic the elastic modulus of different types of soft tissue. The mechanical properties of a variety of A-B-A type gels have been extensively evaluated in the literature [11-16]. A triblock copolymer gel serves as an excellent starting point for a surrogate material, because these gels exhibit predictable mechanical behavior over a known temperature range, and the mechanical properties are relatively stable over time. Further, the gel mechanical properties may be tuned by changing solvent selectivity, triblock copolymer composition, and temperature.

Techniques such as quasi-static compression tests and dynamic torsion tests can be used to evaluate the mechanical properties of gels on the macro-scale, and instrumented indentation [17] may be utilized to measure the mechanical properties of these materials at the micro-scale. The size scale of the indenter is adequate for investigating heterogeneity on the order of microns [18, 19]. Instrumented indentation has been used to characterize quasi-static and dynamic properties of thermoset polymers and elastomers [20, 21]. The capabilities of these indentation systems include the resolution of displacements below one nanometer and forces below one micro-Newton, with measurements on materials possessing an elastic modulus above 1 MPa readily achieved. In the case of polymeric materials, the tip-sample interaction is of critical importance [22] because polymers exhibit strain rate sensitivity and time-dependent mechanical phenomena such as creep and stress relaxation.

Use of instrumented indentation for evaluating tissue surrogates is advantageous because the surface compression loading is similar to that of a blunt impact into soft tissue. Also, current instrumented indentation testing machines permit the exploration of both strain rates, stress states, and resolution of measurement not achievable utilizing conventional mechanical tests, although the current methodologies for modeling tip-sample interactions are limited [23, 24]. Significant challenges exist when conducting measurements on materials with an elastic modulus below 1 MPa using contact diameters below 1 micron. Adhesion between the indenter tip and sample surface affect the governing displacement-based Hertzian contact area approximation used to determine the contact area which, in turn, affects the mechanical property calculations.

In this work, the mechanical behavior of a traditional BG is compared to two different physically associating gels. A methodology is described to conduct surface mechanical property measurements, using instrumented indentation, on soft gel systems (< 1 MPa). Second, a comparison is made using both bulk and surface measurement techniques to highlight the similarities and differences between the gels at differing length scales and frequency regimes.

THEORY

The governing equations for determining the quasi-static or dynamic modulus of elasticity from compression and torsion tests are readily available in the literature [25]. For a cylindrical specimen, the average engineering stress is computed as the load divided by the cross-sectional contact area and the engineering strain is the ratio of change in specimen length to original length. The modulus values reported in this work are the ratio of the average engineering stress to engineering strain in the elastic regime.

To convert indentation loading data into a modulus value, a relation for a cylindrical flat punch from contact mechanics [18] is used;

$$E^* = \frac{P}{2rh}, \tag{1}$$

where E^* is the reduced modulus of the indented material, r is the cylindrical radius of the indenter, and h is the displacement into the surface. The reduced modulus is defined as

$$E^* = \left[\frac{1-\nu^2}{E} + \frac{1-\nu_i^{\,2}}{E_i} \right]^{-1}, \tag{2}$$

where ν and E are the Poisson's ratio and elastic modulus of the indented material, respectively, and the subscript i refers to properties of the indenter. If the indenter tip is taken to be perfectly rigid, and the material is purely isotropic, incompressible, and an elastic response is assumed, the indented material modulus becomes

$$E = \frac{3P}{8rh}. \tag{3}$$

Note that this equation is only valid for a cylindrical punch. Relationships for other geometries, i.e. spherical or conical tips, will have different relationships between modulus, load, and displacement, and will also include a continuously changing contact area, which is often difficult to accurately predict for very soft materials without optically measuring contact area.

For indentation with a flat punch, dynamic storage and loss moduli are calculated in the following way. Assuming isotropic, elastic and incompressible contact, storage modulus is a function of the dynamically measured stiffness *S* (or an instantaneous ratio of change in load to displacement) and is given by

$$E' = \frac{3S}{8r}. \tag{4}$$

This is essentially identical to Equation (3), except that stiffness is measured dynamically. The loss modulus is a function of the measured damping, *C*, associated with the contact, the excitation frequency, ω, and is given by

$$E'' = \frac{3\omega C}{8r}. \tag{5}$$

EXPERIMENTAL[‡]

BG samples were made from a 20% mass fraction of 250 bloom type A ordnance gelatin dissolved into 40 °C ultra-pure filtered water. Right cylinder compression specimens were fabricated by pouring the solution into an open-faced aluminum mold and cooling to ambient conditions. The diameter and length of the specimen geometry is 50.8 mm and 12.7 mm, respectively, and thus yields a length to diameter ratio of four.

Physically associating gels were made from two commercially available triblock copolymers consisting of polystyrene (PS) and polyisoprene (PI). Both triblock copolymers consisted of 80 % mass fraction triblock (PS-PI-PS) and 20 % mass fraction diblock (PS-PI) composition. One of the gels contained 15 % by mass PS (PS-15) and the other gel contained 30 % by mass PS (PS-30), and was used as received. The polymer was mixed with mineral oil at a ratio of 20 % by volume polymer and 80 % by volume mineral oil. The solution was placed in a nitrogen-purged vacuum oven at ~150 ºC and fully dissolved over a period of about 6 hours. The melt was then poured onto a flat surface where it was allowed to cool and gelate, with the PI chains bridging aggregate PS crosslinks. Both materials were visually transparent and contained no detectable bubbles in their final state.

The mechanical properties of the BG, PS-15 and PS-30 were tested on the bulk scale using an Advanced Rheometer 2000 (TA Instruments) and a servo-hydraulic Instron 1331 mechanical testing frame (MTS Systems). The rheometer has a force resolution of about 10 µN and a displacement resolution of about 1 µm, and is capable of both compression and tension testing. A 4.44 kN capacity load cell was mounted onto the Instron to reduce the signal to noise ratio when testing soft materials. The force resolution of the load cell is about 0.5 mN and displacement resolution of 3 µm.

BG was tested at (10 ± 0.1) °C in compression with the Instron machine, at controlled strain rates of 0.001 s^{-1} and 0.01 s^{-1}. Approximately twenty different samples comprised the data set. Compression tests for PS-15 and PS-30 were performed on the rheometer at room temperature using 12 mm diameter cylindrical specimens that had a thickness of approximately 3 mm. Tests were conducted with displacement rates of (5, 10, 50 and 100) µm/s, yielding equivalent strain rates of approximately (0.0017, 0.0033, 0.0167 and 0.0333) s^{-1}, respectively, until a total of 500 µm displacement occurred. For compression testing of BG, PS-15 and PS-30, the contacts between the machine and sample were well lubricated during testing (with either olive oil or mineral oil) to ensure a uniaxial stress state and to break adhesive effects. To further investigate the mechanical properties of these three materials, dynamic tests were performed on the rheometer within the temperature range of (0 to 50) ºC in torsion using oscillation frequencies of (0.1 and 10) Hz.

PS-15 and PS-30 mechanical properties were measured at the micro-scale using a TriboScope depth-sensing indenter (Hysitron, Inc.). Data is not reported on BG with the indenter due to non-repeatability of tests. During indentation, a tool is pressed into the material surface. Load and displacement is continuously measured during material loading and unloading at resolutions in the sub-µN and sub-nm range, respectively. For all tests performed in this work, a 250 µm radius flat punch indenter was used. The material surface was manually located by manually lowering the tip until an adhesive tensile force was sensed (a negative force). The indenter was lowered until the load reading became zero, as illustrated in Figure 1. Quasi-static indentation tests to a maximum of 600 nm were carried out at (1, 10, 25, 50, 100, 150 and 200) µN/s loading rates. Unloading rates

were approximately the same for each test and holding times were less than four seconds. Data was collected using active load control, meaning the spring force was subtracted from the measured load in real time to achieve true loading rates. Dynamic tests were performed at depths of approximately 500 nm into the sample surface with oscillation amplitudes of about 15 nm. Oscillation frequency was varied from (10 to 200) Hz.

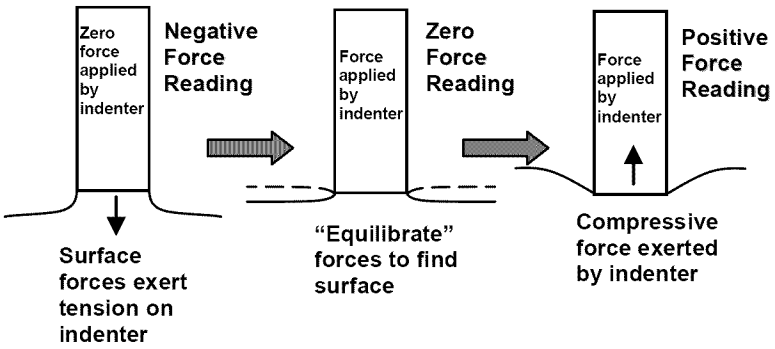

Figure 1: Evolution of measured forces between the indenter and gel in determining point of contact and during loading.

RESULTS AND DISCUSSION

Bulk compression measurements at ~10 ºC indicate BG modulus values of (96 ± 12) kPa at a strain rate of 0.001 s^{-1} and (124 ± 4) kPa at a strain rate of 0.01 s^{-1} to ultimate strains of about 1. The dependence of modulus on strain rate suggests that BG has a significant viscoelastic component to its mechanical behavior. Dynamic measurements from rheometer experiments confirm this as well. At 0.1 and 10 Hz excitation frequencies, the corresponding loss modulus values were calculated to be approximately 20 % of the storage modulus at 10 ºC, as seen in Figure 2. Storage modulus was about 150 kPa while the loss modulus was about 30 kPa. A major weakness of BG, as shown in Figure 2, is at the loss of the gelatin structure between (25 to 30) ºC over a period of time, making testing at room temperature unreliable. Thus, the optimal replacement material for BG should have comparable storage and loss properties that are retained at ambient temperatures.

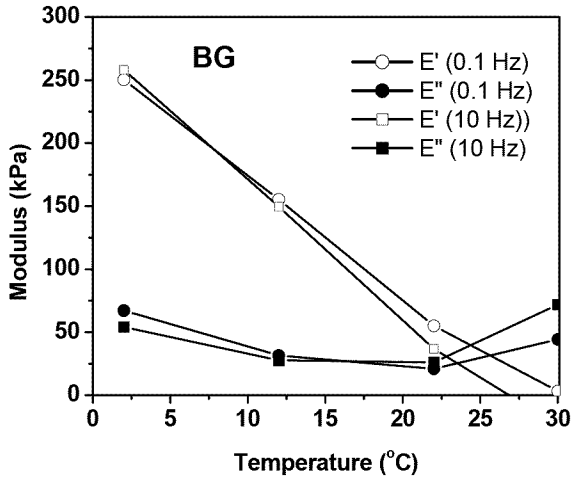

Figure 2: Storage and loss modulus properties for BG as measured dynamically at 0.1 and 10 Hz with the rheometer from 0-30 ºC. Properties vary greatly as a function of temperature.

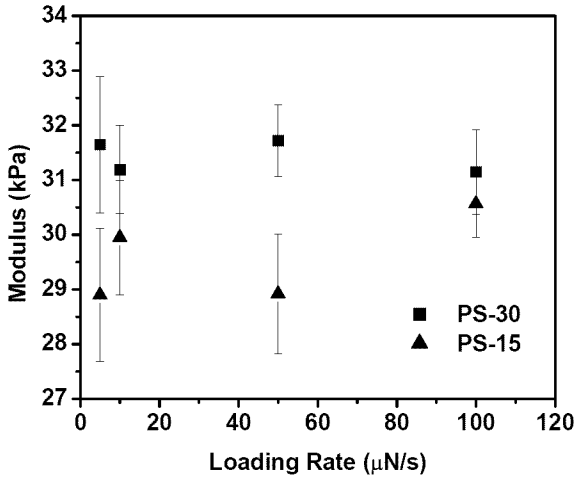

Figure 3: Compression test data for PS-15 and PS-30 at loading rates of (5, 10, 50 and 100) μm/s. Error bars show one standard deviation.

Gel compression tests at room temperature for both PS-15 and PS-30 showed virtually no rate dependence in the range of (5 to 100) μm/s that was considered. Figure 3 shows the averaged modulus data along with standard deviations for ten different runs. For all tests, the average modulus value for PS-15 is lower than that for PS-30; however, for two of the loading rate conditions, the values are within the standard deviations of the measurements. It is expected that PS-15 should have a slightly lower modulus than PS-30 because of the

greater mass fraction of PS. As compared to BG, the synthetic gel modulus values are significantly lower (by a factor of 4 to 5). Dynamic measurements, shown in Figure 4, agree with the compression test data trends at room temperature. The complex modulus values of PS-15 and PS-30 are approximately 18 kPa and 30 kPa, respectively. It is unclear why such a detectable modulus difference exists using the dynamic method as compared to quasi-static uniaxial compression, although the two techniques agree better for the PS gels than for BG. Slippage between contacts or a lack of thermal equilibrium in the material are potential sources of error. Unlike BG, PS-15 and PS-30 have fairly stable mechanical properties in the range of (0 to 50) °C, which make their properties easy to assess at room temperature.

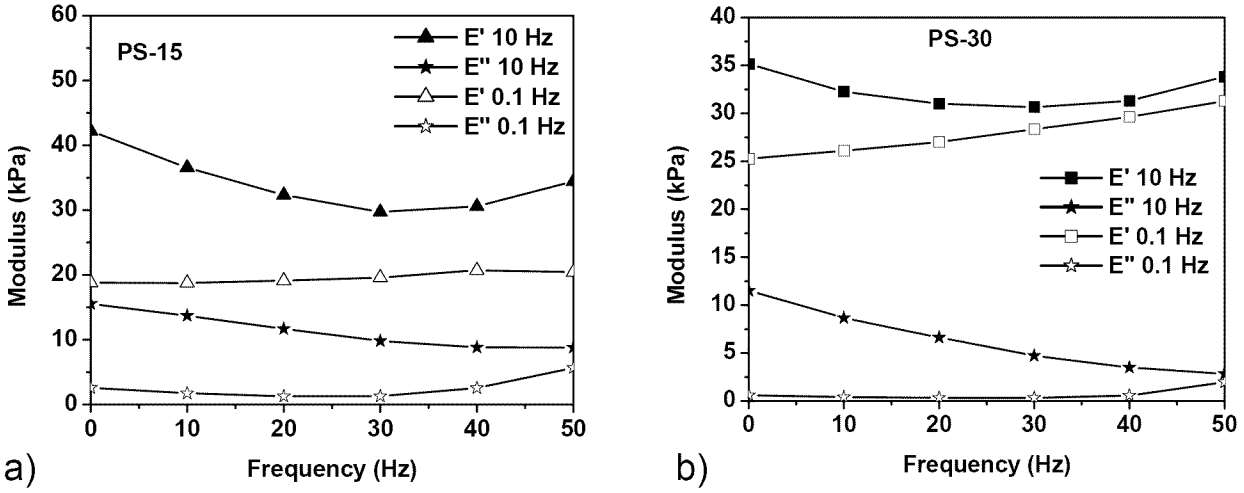

Figure 4: Storage and loss modulus properties for PS-15 (a) and PS-30 (b) measured dynamically at 0.1 and 10 Hz with the rheometer in the temperature range of 0-50 °C.

Indentation measurements were conducted to compare micro-scale mechanical properties to those measured at the macro-scale. Quasi-static indentation tests on PS-15 and PS-30 yielded a rate-dependent hardening response that was not seen with the compression tests. For both gels, a higher loading rate yielded a higher load at the same displacement into the surface. An example of this effect is seen in Figure 5. Only the linear portions of the loading curves in Figure 5 were considered for modulus measurements (typically from approximately 100 nm to 600 nm of indentation depth). The non-linear, initial portions of the loading curve are likely due to inertial ef-

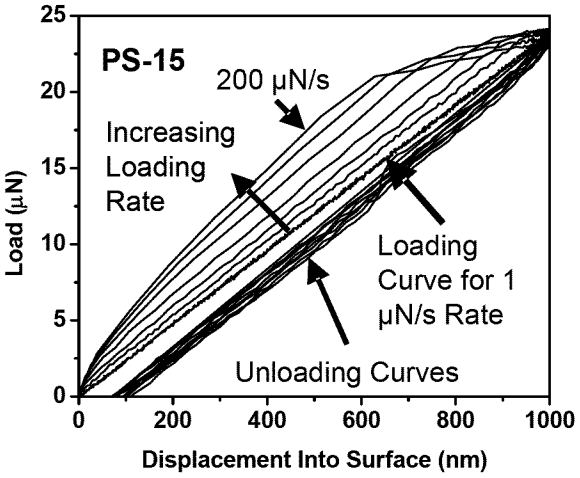

Figure 5: Loading and unloading curves for loading rates of (1, 10, 25, 50, 100, 150 and 200) μN/s on PS-15, illustrating the time-dependent response of the material. Results were similar for PS-30.

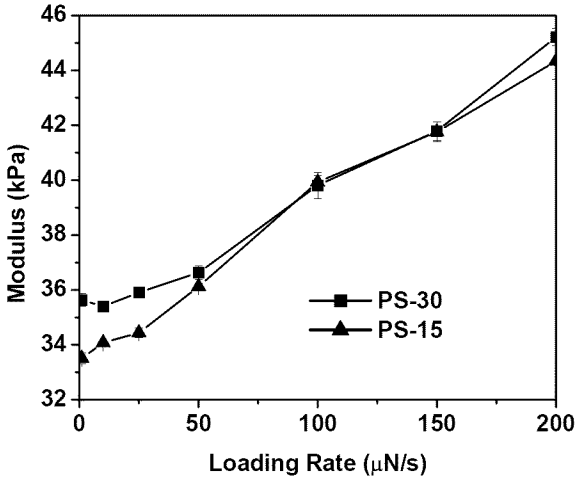

Figure 6: Indentation modulus of PS-15 and PS-30 as a function of loading rate. Error bars represent one standard deviation in the measurements.

-fects within the gel. If the assumptions made are correct, the *P* versus *h* plot should be linear for a flat punch. Therefore, the slope of the line is critical to determining the measured modulus, while the origin of the fit will not influence the measured modulus that requires a fit to both the load and the intercept. This is not the case with other indenter geometries such as spherical, Berkovich, conical, or cube corner.

Modulus values for PS-15 and PS-30 as a function of loading rate are reported in Figure 6. Interestingly, the modulus of PS-15 is lower than that for PS-30 at loading rates below 50 μN/s and above this rate the modulus values are within the standard deviation of each other (~1 kPa). It is possible that at such high loading rates this behavior is an artifact of the machine's collection capabilities, or it could point to the fact that the mobility for each material becomes similar at higher loading rates. For stress rate comparison, the stress rate during indentation loading at 50 μN/s was similar to the rheometer loading rate of 10 μm/s.

The major noticeable difference between the quasi-static compression data and the indentation data for PS-15 and PS-30 is the evidence for rate dependence in the materials. Although the average applied stress during indentation is much less than for compression tests, the stress field underneath the flat punch indenter is much different than that for uniaxial compression. Uniaxial compression assumes an evenly distributed stress state in the direction of loading. However, at the edges of the flat punch, stresses are very large (theoretically infinite). Therefore, this highly-stressed annular boundary area, which is not present in compression, may contribute to the rate dependence of the two materials. Likewise, the modulus measured by the indentation technique is also higher than that for compression.

The average storage modulus of both PS gels was (47 ± 8) kPa and the loss modulus was (2.4 ± 1.0) kPa when the data was averaged over the frequency range of (10 to 200) Hz, as measured by dynamic indentation. These averages yield complex modulus values only slightly higher than those found using the quasi-static indentation technique, which could be due to heating effects at higher frequencies. However, the measured storage and loss modulus varies quite a bit over the frequency range as shown in Figure 7. At 10 Hz, both materials had a storage modulus of 39 kPa, which is still higher than values determined using the rheometer, but are within close agreement of the quasi-static data. This discrepancy between indentation storage modulus and the rheometer values may be due to the fact that the indentation measurements were dependent on the calibrated dynamic response of the indentation instrument, which has a much greater stiffness than that of the contact. Similar to the trend with the loss modulus measured via the rheometer, there is a consistent increase in its magnitude as a function of frequency. As measured with indentation, the loss modulus is (0.20 ± 0.03) kPa at 10 Hz and (3.7 ± 0.1) kPa at 200 Hz for both PS-15 and PS-30. The loss modulus was 0.5 % of the storage modulus at 10 Hz and 16 % at 200 Hz.

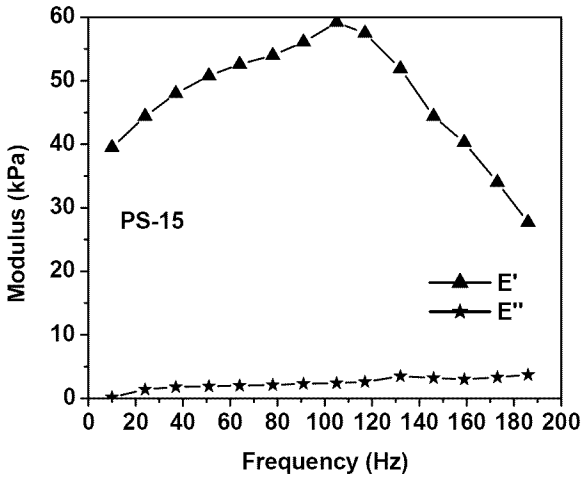

Figure 7: Storage and loss modulus of PS-15 as measured dynamically with the flat punch indenter over a range of 10-200 Hz. Results for PS-30 were similar.

CONCLUSIONS

BG modulus was successfully measured at 10 ºC using quasi-static and dynamic techniques on the macro-scale. Two physically associating gels, PS-15 and PS-30, based on swollen triblock copolymers, were found to exhibit quasi-static properties within the range of BG. Although the modulus values of PS-15 and PS-30 were lower than BG, they were still at the lower end of the modulus range reported for soft tissue (~25 kPa). Even though only two specific gels were studied in this work, the gel systems may be tailored to have their modulus increased. Compared to BG, both PS-15 and PS-30 were found to have a greater degree of thermal stability. BG gels have a shelf life of just a few days at ambient conditions. A successful methodology was developed for mechanically characterizing materials with a modulus below 1 MPa using instrumented indentation, which applies to a number of other soft material systems. Both macro and micro-scale tests of PS-15 and PS-30 yielded similar modulus measurements. Macro-scale measurements found little rate dependence on either PS-15 or PS-30, while micro-scale measurements revealed rate dependent behavior. The reason for this difference was thought to be due to the boundary conditions present during flat punch indentation as compared to a compression test. Interpretation of the dynamic mechanical properties of the two gels measured with instrumented indentation and comparisons to the loading rate effects were difficult. More extensive work on these and other similar material systems is planned for the future.

ACKNOWLEDGEMENTS

Thanks is given to Dr. Peter Drzal at PPG Industries, Pittsburgh, for his insight and comments on this publication. This research was supported in part by TFJ's appointment to the Postgraduate Research Participation Program at the U.S. Army Research Laboratory administered by the Oak Ridge Institute for Science and Education.

‡ Certain commercial equipment and materials are identified in this paper in order to specify adequately the experimental procedure. In no case does such identification imply recommendations by the Army Research Lab and National Institute of Standards and Technology nor does it imply that the material or equipment identified is necessarily the best available for this purpose.

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